High-Speed Movies at the Nanoscale

CONVENTIONAL transmission electron microscopy is an extremely powerful tool for revealing the structure and properties of materials at atomic, nanometer, and micrometer scales; nonetheless, the tool has its limitations. For instance, transmission electron microscopy is not fast enough to measure rapidly evolving nano- and microscale processes that occur over very short timescales.

With the R&D 100 Award–winning movie-mode dynamic transmission electron microscope (MM-DTEM), scientists can now capture a sequence of nine images or diffraction patterns over a period of 1 to 100 microseconds. The resulting nine-frame movies provide details never before seen of basic material and biological processes, enabling an entirely new class of experiments and dynamic measurements.

Window to the World of the Very Small

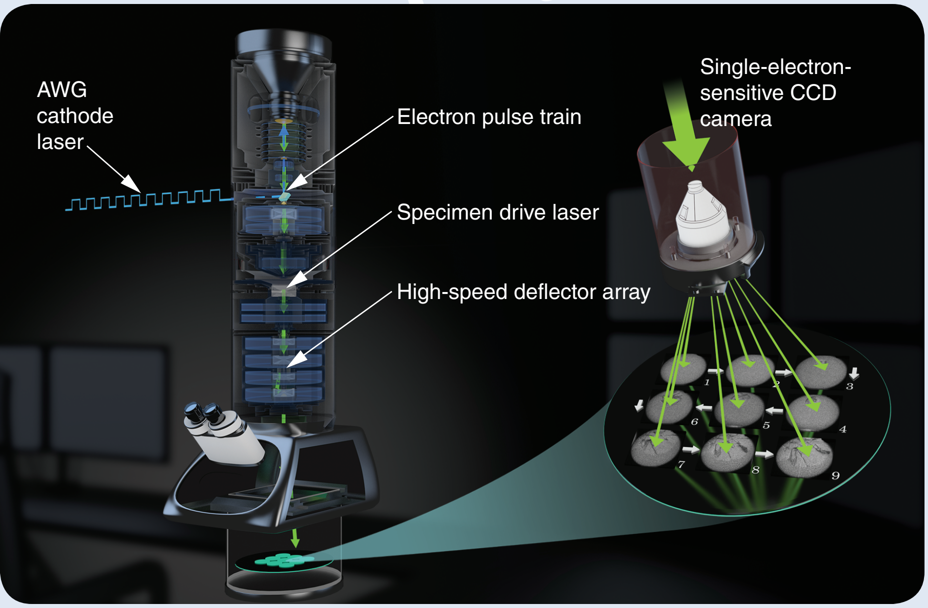
The Livermore MM-DTEM team led by Thomas LaGrange and Bryan Reed also includes Glenn Huete, Richard Shuttlesworth, and William DeHope. The product is marketed through Integrated Dynamic Electron Solutions, Inc., which also markets a product based on the precursor system called the single-shot DTEM. This earlier microscope, which won an R&D 100 Award in 2008, captures a single snapshot of a rapid process with nanosecond exposure times. (See *S&TR*, October/November 2008, pp. 4–5.) MM-DTEM changed the game with its ability to record multiple images of a fast-evolving material process. The product is ideally suited for directly observing complex processes such as microstructural changes, phase transformations, and chemical reactions. "These are the processes that govern the fabrication and real-world performance of nanostructured materials," says LaGrange.

The product could also be used to analyze little-understood biological processes, including host–pathogen interactions and protein–protein binding. For instance, MM-DTEM could capture conformational changes in molecules during the protein-binding process, allowing researchers to see how protein molecular structure evolves during binding, a process critical to biological function. (See *S&TR*, September 2013, pp. 4–11 for early applications of MM-DTEM.)

Lights, Camera, Action!

MM-DTEM is based on a transmission electron microscope modified to include two pulsed lasers—a sample drive laser and an arbitrary waveform generation (AWG) cathode laser.

The MM-DTEM development team: (from left) Thomas LaGrange, Glenn Huete, William DeHope, Richard Shuttlesworth, and Bryan Reed.



With the movie-mode dynamic transmission electron microscope (MM-DTEM), researchers can now capture the details of material processes that evolve over nanosecond timescales. This artist's conception of MM-DTEM shows the arbitrary waveform generator (AWG) cathode laser that produces precisely shaped and timed electron pulses, the high-speed pulse deflector that is synchronized with the AWG laser, and the high-resolution charge-coupled-device (CCD) camera that records the resulting array of images or diffraction patterns.

(See the figure above.) The sample drive laser releases a short (about 12-nanosecond-long) pulse that heats a small region of the specimen, initiating a desired process.

After an electronically controlled time delay, the AWG cathode laser emits a train of precisely shaped laser pulses. Each pulse strikes a photocathode, creating, in turn, an electron pulse with the same temporal shape as the initiating laser pulse. This AWG cathode laser is the heart of the system's innovation, providing a flexible platform that can create electron pulses lasting from 10 nanoseconds to 1 microsecond, and frame-to-frame spacing ranging from 50 nanoseconds to 150 microseconds.

Each electron pulse is then accelerated to 70 percent the speed of light and focused onto a specimen that is 5 to 250 nanometers thick. The electrons interact and pass through the material to lenses, which produce either diffraction patterns or real-space photographic images on a charge-coupled-device camera. A high-speed deflector array, precisely synchronized with the AWG cathode laser, deflects each pulse to a different region on the camera. The end result is a series of images that form the frames for a movie. The current system uses a 3-by-3 image array with 150-nanosecond frame-to-frame times and exposure intervals as short as 10 nanoseconds.

The team plans to expand the system's capabilities. For instance, with only minor upgrades, the system could produce a 5-by-5 array and take movies with even shorter interframe times. A new camera system under consideration would supply images of even higher resolution. "We're looking at using a direct-electron-detection camera, which would not depend on scintillators, is less noisy, and is more sensitive by a factor of 10," says LaGrange. "Because this camera records electrons directly, we would be able to use a lower-dose electron beam, which is preferable for imaging biological samples."

The team is also developing methods for exploring different processes in a variety of environments. "Heat from a laser pulse isn't the only way to initiate a process," says Reed. "For example, microelectromechanical system devices can rapidly apply high stress to a sample, deforming it. We could then examine high strain-rate deformation, defect formation, and other interactions that lead to material failure."

It's a Wrap

With frame rates over 100,000 times faster than those of conventional techniques, MM-DTEM is the only system that captures the fine-scale details of rapidly evolving nano- and microscale processes, for both organic and inorganic specimens. "With MM-DTEM, we're opening a new window to the world of small-scale, rapid processes," says LaGrange. "For the first time, researchers will be able to capture a tremendous range of fundamentally and technologically important processes and see the crucial in-between moments of material dynamics."

With the tiny worlds of nanomaterials looming larger in importance every day, tools are needed to record objects and structures at this level as they form, move, and interact. MM-DTEM now makes this nanoscale movie-making a reality.

—Ann Parker

Key Words: arbitrary waveform generator (AWG) cathode laser, electron pulse train, movie-mode dynamic transmission electron microscope (MM-DTEM), R&D 100 Award.

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